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**NEW APPLICATION TRANSMITTAL**

Transmitted herewith for filing is the patent application of:

Inventor(s):

For (title):

**Enclosed are:**

- ☒ 1 sheets of drawing(s). ☐ informal ☐ formal  
☒ An assignment of the invention to Bechtel BWTX Idaho, LLC  
☐ A certified copy of a \_\_\_\_\_ application.  
☐ An associate power of attorney.  
☒ Combined Declaration and Power of Attorney.  
☐ Information Disclosure Statement.  
☐ Form PTO-1449 (together with citations).

**Fee Calculation (37 CFR 1.16)**

CLAIMS AS FILED

| NUMBER FILED  | NUMBER<br>EXTRA | RATE      | BASIC FEE<br>\$690.00 |
|---|-----------------|-----------|-----------------------|
| Total<br>Claims (37 CFR 1.16(c)) 15 -20=                | 0               | X \$18.00 | = 0                   |
| Independent<br>Claims (37 CFR 1.16(b)) 3 -3=            | 0               | X \$78.00 | = 0                   |
| Multiple dependent claim(s), if any<br>(37 CFR 1.16(d)) |                 | \$260.00  | = 0                   |
|   |                 |           | TOTAL \$ 690.00       |

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☒ Any additional filing fees required under 37 CFR 1.16(a), (f) or (g) (filing fees).  
☒ Any additional filing fees required under 37 CFR 1.16(b), (c) and (d) presentation of extra claims.)  
☐ Any additional filing fees required under 37 CFR 1.16(e) (surcharge for filing the basic filing fee and/or declaration on a date later than the filing date of the application).  
☐ Any patent application processing fees under 37 CFR 1.17.  
☐ The issue fee set in 37 CFR 1.18 (at or before mailing of Notice of Allowance, pursuant to 37 CFR 1.311(b)).

*W. Gary Goodson*

**SIGNATURE OF ATTORNEY**

W. Gary Goodson

Reg. No. 22.387

**A PROCESS FOR PRODUCING BIODIESEL, LUBRICANTS, AND FUEL**  
**AND LUBRICANT ADDITIVES IN A CRITICAL FLUID MEDIUM**

**CONTRACTUAL ORIGIN OF THE INVENTION**

5           The United States has rights in this invention pursuant to Contract No. DE-AC07-94ID13223 between the U.S. Department of Energy and Lockheed Martin Idaho Technologies Company.

**RELATED APPLICATION**

10           This application claims priority from provisional application S/N 60/094,076 filed July 24, 1998.

**BACKGROUND OF THE INVENTION**

**Field of the Invention**

15           The present invention relates to a process for producing biofuels as alternatives or additives to currently used petroleum-based automotive or other vehicular fuels and lubricants by reacting fats and oils such as triglycerides and free fatty acids in a single critical phase to provide increased reaction rates and decrease the loss of catalyst or catalyst activity. The invention includes the transesterification of triglyceride-containing  
20           substances and esterification of free fatty acid-containing substances with alcohol to produce alkyl esters of triglycerides, a desirable additive or alternative for petroleum diesel fuel or lubricants.

**Description of the Prior Art**

25           Significant quantities of esters such as triglycerides and free fatty acids are available from inexpensive feedstocks such as, animal fats, vegetable oils, rendered fats, restaurant grease and waste industrial frying oils. The triglyceride esters can be reacted, or transesterified, with alcohol to produce glycerol and the alkyl esters, and the free fatty acid can be reacted, or esterified, with alcohol or water to produce the alkyl ester. These  
30           alkyl esters create desirable additives or alternatives to petroleum diesel fuel as well as

other high value end products such as detergent surfactants, herbicides, pesticide diluents, sticking agents, or lubricating additives for hydraulic and transmission fluids to name a few. Consequently, numerous patents exist dealing with processes surrounding transesterification of triglycerides and esterification of free fatty acids with alcohols such as methanol, ethanol or butanol to create the corresponding alkyl esters. U. S. Patents 5,713,965 and 5,525,126, incorporated herein by reference, are examples of such processes.

The transesterification or esterification reaction is normally carried out in an excess of the stoichiometric quantity of alcohol and a catalyst, usually a base such as potassium hydroxide although, the reaction can also proceed with an acid catalyst as well. In addition to creation of the alkyl esters, the transesterification reaction also produces glycerol.

Traditionally, triglyceride transesterification requires a multiple step process with one or more batch reactors. Initially, the triglycerides and alcohol form two immiscible liquid phases. As the reaction proceeds, two separate liquid phases form. One contains the newly formed alkyl esters of the triglyceride and the other the glycerol with the excess alcohol, catalyst and feed oil being dispersed into both phases. The reaction time for each step typically is measured in hours and once completed the liquid products must be allowed sufficient time to separate phases before additional processing and separation steps can occur to produce the final products. Excess alcohol must be recycled and the unused catalyst typically must be neutralized.

Even the most efficient of the traditional processes require multiple hours to process each batch of feed. Additionally, significant problems arise in the separation steps. Significant quantities of glycerol left in the alkyl esters diminishes the quality of the diesel fuel and likewise contaminated glycerol also loses much of its value compared to pure uncontaminated glycerol. Traditionally the separation procedures necessary to adequately clean the two product streams produces large quantities of waste water thereby creating additional cost and/or process complexity.

**SUMMARY AND OBJECTS OF THE INVENTION**

This invention provides a single-phase process for producing alkyl esters useful as biofuels and lubricants by the reactions of triglyceride esters and free fatty acids. This invention teaches a process where the reactants enter a reactor, whether batch or continuous, dissolved in a critical fluid. The critical fluid provides a single-phase medium in which diffusion of the reactants into different liquid phases is eliminated, and mass transfer limitations are essentially eliminated thereby increasing the overall reaction rate. A critical fluid is a fluid whose temperature is within 20% of the critical temperature of the fluid as measured in Kelvin and pressure within 0.5-1.5 times the critical pressure as modified by any co-solvent.

Additionally, the solubility of the reaction products such as alkyl esters in the critical fluid can be controlled by controlling the reactor's temperature and pressure. Where a reaction product's solubility is low or nonexistent such as glycerol it drops out of the fluid as it is created, thereby driving the reaction equilibrium toward product production which significantly reduces the quantity of excess reactants such as alcohol needed to drive the reaction to completion.

The use of a critical fluid also allows for a wide range of catalysts, both liquid phase and reusable solid phase acid or base catalysts. Solid phase catalysts have significant additional advantages by limiting unwanted side reactions and producing higher conversion rates of the desired products.

With the reaction completed, the critical fluid medium also facilitates clean, efficient separations. The reaction products typically can be sequentially and selectively removed from the critical fluid medium by adjusting the temperature and pressure of the critical fluid medium. In a transesterification reaction of triglycerides, the glycerol is removed first leaving the alcohol esters in the critical fluid. With the glycerol removed, the temperature and pressure is again changed to drop out the alkyl esters. Once the products are removed the critical fluid and any excess reactants are returned to the beginning of the process.

It is the object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances capable of completing the reaction in significantly less time than present conventional processes.

5 It is another object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances capable of using reusable catalyst thereby avoiding the need for process neutralization.

It is yet another object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances capable of using a solid phase catalyst.

10 It is yet another object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances with higher yields of desired product over conventional processes.

15 It is yet another object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances capable of operating in a single phase, thereby eliminating immiscible liquid phases and reducing mass transfer reaction limitations.

It is yet another object of the present invention to provide a process for reacting triglyceride- and free fatty acid- containing substances with improved separation capabilities for separating purified reaction products without the need for washing steps.

20 It is yet another object of the present invention to provide a process for reacting triglycerides and free fatty acids which can quantitatively react the triglycerides or free fatty acids with significantly less excess alcohol than present processes.

#### **BRIEF DESCRIPTION OF THE DRAWINGS**

25 In order that the manner in which the above-recited and other advantages and objects of the invention are obtained, a more particular description of the invention briefly described above will be rendered by reference to specific embodiments thereof which are illustrated in the appended drawings. Understanding that these drawings depict only typical embodiments of the invention and are not therefore to be considered

to be limiting of its scope, the invention will be described and explained with additional specificity and detail through the use of the accompanying drawings in which:

Figure 1 is a simplified flow diagram which illustrates an embodiment of the invention employing a continuous reactor.

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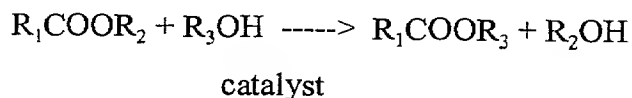
### **DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS**

The present invention describes a process for reacting organic compounds having the generic formula  $R_1COOR_2$  with short-chain ( $C_1$  to  $C_4$ ) alcohols in a single critical fluid phase medium over an acidic or basic catalyst to produce alkyl esters suitable as a biodiesel fuel. One skilled in the art will recognize that organic compounds from the family of compounds known as acylglycerols, fats, oils, waxes, or free fatty acids have the general formula  $R_1COOR_2$ . One skilled in the art will also recognize that acylglycerols can be mono, di, or tri substituted in any manner. The invention includes the transesterification and esterification of  $R_1COOR_2$  molecules with short-chain alcohols such as methanol, ethanol, propanol, or butanol to produce alcohol esters. The invention also encompasses the acid and base catalyzed hydrolysis of  $R_1COOR_2$  compounds in a single critical fluid medium where  $R_1$  is a chain of aliphatic hydrocarbons ( $C_4$  to  $C_{36}$ ), and where  $R_2$  can either be glycerol or can be another aliphatic hydrocarbon chain ( $C_4$  to  $C_{36}$ ) linked to  $R_1$  via the carboxylic ester ( $BCOO-$ ) linkage. The reaction is generally described as follows:

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In the case of mono-, di-, and triglycerides, fats and oils,  $R_1$  is the aliphatic hydrocarbon ( $C_4$  to  $C_{24}$ ) chain,  $R_2$  is glycerol and  $R_3$  is a hydrocarbon group, and preferably short chain alkyl group, more preferably methyl, ethyl, propyl, or butyl group attached to the alcohol. In the case of free fatty acids,  $R_1$  is the aliphatic hydrocarbon chain wherein the terminal carboxylic acid group could be  $R_1COO^-$ , or  $R_1COOH$ , or  $R_1COO^- M^+$  where  $M^+$  is a metal (thus, the salt of a fatty acid); and,  $R_3$  is the short-chain hydrocarbon attached to the alcohol. In the case of a wax,  $R_1$  is an aliphatic hydrocarbon chain linked to  $R_2$  via

25

the carboxylic ester linkage, and  $R_3$  is the short-chain hydrocarbon attached to the alcohol.

Figure 1 provides a basic flow diagram for the glyceride reaction process employing a continuous reactor. Throughout the description of the process diagram, the various process vessels will be numbered between 1 and 99, with the various process streams being numbered beginning with 100. While the following discussion will describe a continuous reactor process for a transesterification reaction of glycerides with an alcohol ROH, one skilled in the art will recognize the process principles apply equally well in other process settings such as ones using batch reactors and separation processes as well as reactions producing alternate products to the alcohol esters as well as processes beginning with fatty acid feeds.

A glyceride containing feed **100** is mixed with an input alcohol stream **102**. The choice of alcohol will be a function of the desired reaction product, and typically such alcohols as methanol, ethanol, propanol and butanol are chosen for practical reasons, however, one skilled in the art will readily recognize the flexibility of choices and non-limiting aspect of the above list. This input alcohol stream **102** contains approximately a stoichiometric quantity of alcohol necessary to quantitatively react the input glyceride feed **100**. While the reaction will require some excess alcohol, that needed excess is contained in a critical fluid recycle stream **104** which provides a transport medium that solvates the reactants to create the required process conditions. The exact critical fluid employed for a given reaction will depend on specifically chosen process parameters such as temperature, pressure, desired reaction products, solubility of the reaction products, quantity of excess alcohol needed to drive the reaction to completion, post reaction separation processes and chosen catalyst. Examples of possible critical fluid solvents are carbon dioxide, sulfur dioxide, methane, ethane, propane, or mixtures thereof, with or without critical fluid co-solvents such as methanol, ethanol, butanol or water. Naturally, to the extent quantities of the critical fluid are lost during the process they can be replenished with a critical fluid make-up stream **106**.

The mixing of the input feed 100 (substance containing free fatty acids and/or glycerides), the input alcohol 102, the critical fluid recycle 104 and critical fluid make-up 106 streams creates a reactant input stream 108 which is fed into a continuous reactor 10. The temperature and pressure of the reactant input stream will depend on its components and the desired process parameters. The important criteria for the critical fluid is its ability to dissolve the reactants. Reaction temperatures should be within 20% of the critical temperature of the fluid as measured in Kelvin, and pressures within 0.5-15 times critical pressure as modified by any co-solvent. Reaction temperatures are typically in the range from about 20 to 200 degrees C with reaction pressures in the range from about 150 psig to 4000 psig.

The transesterification reaction generally proceeds in the presence of a catalyst, either acidic or basic. Liquid acids and bases, such as the common inorganic acids HCl, H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub> and inorganic bases NaOH and KOH typically provide the needed catalytic activity. Additionally, the use of a critical fluid medium allows for use of a solid phase catalysts with either acidic or basic surfaces such as microporous crystalline solids, such as zeolites, and non-crystalline inorganic oxides such as alumina, silica, silica-alumina, boria, oxides of phosphorus, titanium dioxide, zirconium dioxide, chromia, zinc oxide, magnesia, calcium oxide, iron oxides, unmodified, or modified with chlorine, fluorine, sulfur or an acid or base, as well as mixtures of the above group or an exchange resin with either acidic or basic properties. Where solid catalysts are used in the reactor 10, they may create a catalytic packed bed or float free inside the reactor.

As the reaction proceeds, glycerol and the alkyl esters of the glyceride are produced. Glycerol has low solubility in critical fluids such as CO<sub>2</sub> and propane which will cause the glycerol product to drop out of the critical fluid medium. This removal of the glycerol from the reaction phase of the critical fluid medium will enhance the reaction equilibrium and drive the reaction further to completion with limited excess alcohol. One skilled in the art will then recognize that the quantity of excess alcohol required to drive the reaction to completion will depend not only on such factors as the desired reaction rate, but also the glycerol solubility in the chosen critical fluid.



The ability of the critical fluid medium to solvate the reactants eliminates the immiscible phases found in conventional processes. The single phase reaction eliminates inter-phase mass transfer of the individual reactants and catalyst, thereby greatly increasing the reaction's rate.

5       After completion of the reaction the reaction vessel may be the separation vessel, a final product stream 110 exits the reactor 10 and enters a first product separator 12. In the first product separator 12 the product stream's temperature and/or pressure are modified to allow the least soluble product in the critical fluid to quantitatively drop out, the glycerol in this embodiment. Once the glycerol has dropped out of the critical fluid  
10       medium, a physical separation of the two phases can be readily accomplished. A glycerol product stream 112 and a glycerol depleted product stream 114 exists the first separator 12. The glycerol depleted product stream 112 consists of the critical fluid, excess alcohol, alcohol ester of the glycerides and any remaining catalyst, if a liquid catalyst is used, and then enters a second product separator 14. Again the temperature and  
15       pressure of the critical fluid are lowered to allow the desired product, the alkyl ester of the glyceride of this embodiment, to drop out of the critical fluid while retaining the excess alcohol in the critical fluid. The physical separation of the two phases then creates a second product stream of the alcohol ester 116 and the critical fluid recycle 104 which will be reintroduced back into the front of the process after having its pressure and  
20       temperature restored to the original input reaction requirements.

While this embodiment describes a process with two product streams, the glycerol and alkyl ester, it should also be apparent to one skilled in the art that a reaction producing more than two products can produce multiple product streams by simply increasing the number of product separators.

25       Although the present invention has been described with reference to preferred embodiments, those skilled in the art will recognize that changes may be made in form and detail without departing from the spirit and scope of the invention.

**WE CLAIM:**

1. A process for producing alkyl esters useful in biofuels and lubricants by reacting glyceride- or free fatty acid- containing substances in a critical fluid medium, said process comprising the steps of:

5 (a) dissolving the glyceride- or free acid- containing substance and an alcohol or water input into a critical fluid; and

(b) reacting the glyceride- or free fatty acid- containing substance with an alcohol or water in the presence of a catalyst to produce final products.

10 2. A process as described in claim 1 wherein said alcohol is an ROH input, wherein R is a short chain alkyl group.

3. A process as described in claim 2 where said alcohol is chosen from among the group ethanol, methanol, propanol, butanol, isopropanol and isobutanol.

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4. A process as described in claim 1 wherein said catalyst is a liquid catalyst.

5. A process as described in claim 1 wherein said catalyst is a solid catalyst.

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6. A process as described in claim 4 wherein said liquid catalyst is an acid.

7. A process as described in claim 4 wherein said liquid catalyst is a base.

8. A process as described in claim 5 wherein said solid catalyst is an  
25 inorganic oxide.

9. A process as described in claim 8 wherein said inorganic oxide is from among the group of alumina, silica, silica-alumina, boria, oxides of phosphorus, titanium dioxide, zirconium dioxide, chromia, zinc oxide, magnesia, ion exchange resins, silicate

catalysts, and calcium oxide either unmodified or modified with chlorine, fluorine, sulfur or an acid or base.

10. A process as described in claim 5 wherein said solid catalyst is an ion  
5 exchange resin with either acidic or basic properties.

11. A process as described in claim 1 wherein said process further comprises the steps of:

- 10 (a) separating a glycerol product from said final products; and  
(b) separating an alkyl ester product from said critical fluid.

12. A process as described in claim 11 wherein said process further comprises the step of recycling said critical fluid for use in a later reaction.

13. A process for producing alkyl esters useful in biofuels and lubricants by  
15 reacting a glyceride- containing substance in a critical fluid medium, said process comprising the steps of:

- 20 (a) dissolving a glyceride- containing substance and an alcohol or water input into a critical fluid; and  
(b) reacting the glyceride- containing substance and alcohol or water input in a reactor in the presence of a catalyst to produce final products; and  
(c) separating an alkyl ester product and a glycerol product from the reaction mixture.

14. A process as described in claim 13 additionally comprising the step of  
25 recycling said critical fluid for re-use in this process.

15. A process for reacting fatty acids in a critical fluid medium, said process comprising the steps of:

- (a) dissolving a fatty acid- containing substance and an alcohol input into a critical fluid; and
- 5 (b) reacting the fatty acid- containing substance and an alcohol or water input in a reactor in the presence of a catalyst to produce final products; and
- (c) separating an alcohol ester product and a glycerol product from the reaction mixture.

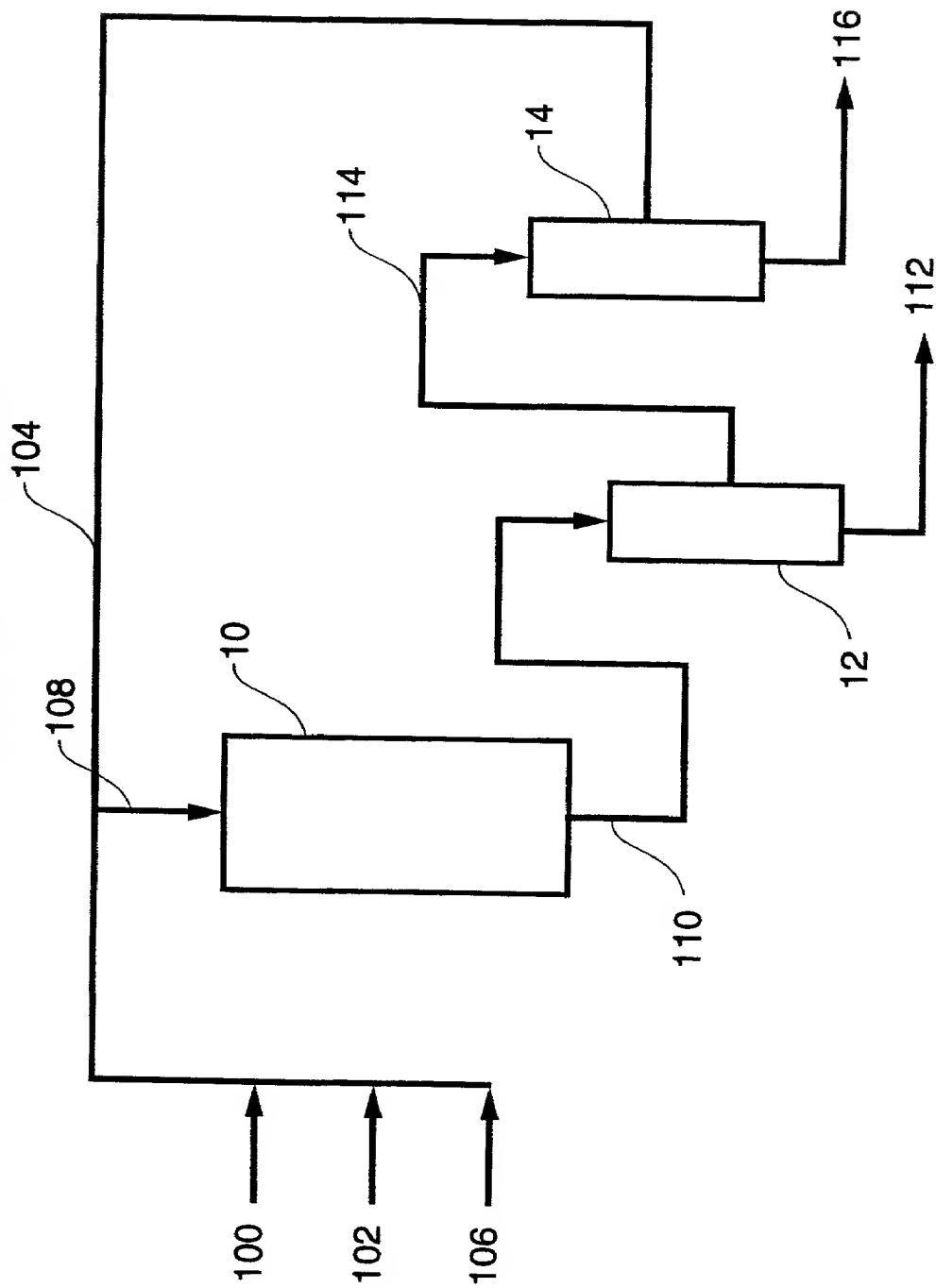


FIG. 1

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|  |                               |   |
|--|-------------------------------|---|
| <b>DECLARATION FOR UTILITY OR DESIGN PATENT APPLICATION</b><br>(37 CFR 1.63) | <b>Attorney Docket Number</b> | LIT-PI-099  |
|  | <b>First Named Inventor</b>   | Dnaiel M. Ginosa  |
|  | <b>COMPLETE IF KNOWN</b>      |   |
|  | <b>Application Number</b>     | 09 / 554,708  |
|  | <b>Filing Date</b>            |   |
|  | <b>Group Art Unit</b>         |   |
| <input type="checkbox"/> Declaration Submitted with Initial Filing           | OR                            | <input checked="" type="checkbox"/> Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required) |
| <b>Examiner Name</b>   |                               |   |

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Process for Producing Biodiesel, Lubricants, and Fuel and Lubricant Additives in a Critical Fluid Medium

the specification of which

(Title of the Invention)

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY)

as United States Application Number or PCT International

Application Number 09/554,708

and was amended on (MM/DD/YYYY)

(if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

| Prior Foreign Application Number(s) | Country | Foreign Filing Date (MM/DD/YYYY) | Priority Not Claimed     | Certified Copy Attached? |                                     |
|-------------------------------------|---------|----------------------------------|--------------------------|--------------------------|-------------------------------------|
|                                     |         |                                  |                          | YES                      | NO                                  |
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☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below.

| Application Number(s) | Filing Date (MM/DD/YYYY) | <input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto. |
|-----------------------|--------------------------|--|
| 60/094,076            | 07/24/98                 |  |

[Page 1 of 2]

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## DECLARATION — Utility or Design Patent Application

I hereby claim the benefit under 35 U.S.C. 120 of any United States application(s), or 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

| U.S. Parent Application or PCT Parent Number | Parent Filing Date (MM/DD/YYYY) | Parent Patent Number (if applicable) |
|--|---------------------------------|--------------------------------------|
| PCT/US99/16669                               | 07/02/99                        |                                      |

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Customer Number

OR

☒ Registered practitioner(s) name/registration number listed below

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| Name            | Registration Number | Name | Registration Number |
|-----------------|---------------------|------|---------------------|
| W. Gary Goodson | 22,387              |      |                     |

☐ Additional registered practitioner(s) named on supplemental Registered Practitioner Information sheet PTO/SB/02C attached hereto.

Direct all correspondence to: ☒ Customer Number or Bar Code Label ☐ Correspondence address below

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor:

☐ A petition has been filed for this unsigned inventor

|  |                     |                        |           |
|--|---------------------|------------------------|-----------|
| Given Name (first and middle (if any)) |                     | Family Name or Surname |           |
| Daniel M.                              |                     | Ginosar                |           |
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| Post Office Address                    |                     |                        |           |
| City                                   | Idaho Falls         | State                  | ID        |
|  |                     | ZIP                    | 83404     |
|  |                     | Country                | US        |

☐ Additional inventors are being named on the 1 supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto

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## DECLARATION

ADDITIONAL INVENTOR(S)  
Supplemental Sheet  
Page 1 of 1

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

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Family Name or Surname

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Fox

Inventor's  
Signature

*Robert V. Fox*

Date

7/24/2000

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Family Name or Surname

Inventor's  
Signature

Date

Residence: City

State

Country

Citizenship

Post Office Address

Post Office Address

City

State

ZIP

Country

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

Given Name (first and middle [if any])

Family Name or Surname

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